भारतीय मानक

IS 17916 : 2022

Indian Standard

टॉल्यूइन डाईआइसोसाईनेट (टीडीआई-80) — विशिष्टि

Toluene Diisocyanate (TDI-80) — Specification

ICS 71.080.99

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Organic Chemicals, Alcohols and Allied Products Sectional Committee, PCD 9

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Organic Chemicals, Alcohols and Allied Products Sectional Committee had been approved by the Petroleum, coal and Related Products Division Council.

Toluene diisocyanate (TDI) is an organic compound with the formula $CH_3C_6H_3(NCO)_2$. There are two isomers of TDI — 2,4-TDI and 2,6-TDI. TDI is marketed as TDI 80/20 (TDI-80) which is a mixture of 80 percent 2,4-toluene diisocyanate (2,4-TDI) and 20 percent 2,6-toluene diisocyanate (2,6-TDI) and also as TDI 65/35 which contains 2,4-TDI and 2,6-TDI isomers in the ratio of 65 : 35, respectively.

TDI-80 is available in two types, Type I (for low acidity) and Type II (for high acidity). TDI-80 is mainly used in the production of polyurethane foam and other polyurethane products including elastomers, synthetic leather, coated fabrics, paints and adhesives.

Toluene diisocyanate is an irritant to the eyes, skin, and respiratory system. Acute exposure can cause irritation of the nose and throat, shortness of breath, choking, coughing, nausea, vomiting, and abdominal pain.

Based on the recommendations of Department of Chemicals and Petrochemicals, Ministry of Chemicals and Fertilizer, the Committee has decided to formulate Indian Standard on Toluene diisocyanate.

Safety instructions to be followed during testing are given in informative Annex G.

The composition of the Committee, responsible for the formulation of this standard is given at Annex H.

For the purpose of deciding whether a particular requirement of this standard is complied with the final value, observed or calculated, expressing the result of a test or analysis shall be rounded off in accordance with IS 2 : 2022 'Rules for rounding off numerical values (*second revision*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

TOLUENE DIISOCYANATE (TDI-80) — SPECIFICATION

1 SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for toluene diisocyanate (TDI-80), which is mixture of 80 percent 2,4-toluene diisocyanate (2,4-TDI) and 20 percent 2,6-toluene diisocyanate (2,6-TDI).

1.2 This standard cover Type I (for low acidity) of toluene diisocyanate (TDI-80).

2 REFERENCES

The following standards contain provisions which through reference in this text, constitute provisions of the standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on these standards are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	*Title*
1070 : 1992	Reagent grade water — Specification (*third revision*)
5299 : 2001	Methods of sampling and tests for dye intermediates (*first revision*)
8768 : 2000	Method of measurement of colour in liquid chemical products platinum - cobalt scale (*second revision*)

3 REQUIREMENTS

3.1 Description

TDI-80 is a colourless liquid of low viscosity above 15°C. In absence of antioxidant, it turns to pale yellow coloured liquid upon coming in contact with air.

3.2 The material shall also comply with the requirements given in Table 1, when tested as prescribed in col 5 and col 6 of Table 1.

4 PACKING AND MARKING

4.1 Packing

The material shall be packed in suitable drums, tankers, ISO containers etc., as agreed between purchaser and supplier.

4.2 Marking

4.2.1 Each container shall be securely closed and shall bear the following information:

a) Name of the material;
b) Name of the manufacturer;
c) Month and year of manufacture;
d) Lot or batch number;
e) Net weight and gross weight; and
f) Any other statutory requirement.

4.2.2 Each container shall bear the minimum cautionary notice worded as under:

KEEP WELL CLOSED.

TOXIC AND AVOID CONTACT WITH SKIN

4.2.3 *BIS Certification Marking*

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act*, 2016 and the Rules and Regulations framed there under, and the products may be marked with the Standard Mark.

5 SAMPLING

5.1 The method of drawing representative samples of the material and the criteria for conformity shall be as prescribed in **4** of IS 5299.

5.2 Criteria for Conformity

For declaring the conformity of a lot to the requirements of all the other characteristics tested on the composite sample, the test result for each of the characteristics shall satisfy the relevant requirement given in Table 1.

Table 1 Requirements for Toluene Diisocyanate (TDI-80)

(*Clause* 3.2, 5.2 and 6.1)

Sl No.	**Characteristics**	**Requirements**	**Methods of Test, Refer to**	
			Annex	**IS**
(1)	(2)	(3)	(5)	(6)
i)	Appearance	Clear liquid without any haze or foreign particle	A	—
ii)	Colour, Pt-Co, *Max*	25	—	8768
iii)	Assay, percent by mass, *Min*	99.5	B	—
iv)	Hydrolyzable Chlorine, percent by mass, *Max*.	0.008 0	C	—
v)	Acidity (*as* HCl), percent by mass, *Max*.	0.004 0	D	—
vi)	Total Chlorine, percent by mass, *Max*	0.07	E	—
vii)	Isomer Ratio (2,4-TDI : 2,6-TDI), percent by mass	(79 – 81.5) : (18.5-21.0)	F	—
viii)	Antioxidant or/and Performance Additives(1)	—	—	

(1) Name of the Antioxidant or/and Performance Additives, their levels and analysis methods shall be provided on mutual agreement between buyer and seller

6 TEST METHODS

6.1 Test shall be conducted as prescribed in col 5 and col 6 of Table 1.

6.2 Quality of Reagents

Unless specified otherwise, 'pure chemicals' and distilled water (*see* IS 1070) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities, which affect the result of analysis.

ANNEX A

[*Table* 1, *Sl No.* (i)]

DETERMINATION OF APPEARANCE (VISUAL INSPECTION PROCEDURE)

A-1 PRINCIPLE

The sample is examined visually to detect that it is a clear liquid and free from any haze or foreign particles in the sample.

A-2 PROCEDURE

Take a clean and dry 500 ml glass bottle. Visually examine the sample to see if any haze or foreign particles are present. It may be necessary to swirl the sample to stir up any material that has settled to the bottom of the sample bottle.

NOTE — Do not expose the sample to moisture before visual inspection as moisture reacts with toluene diisocyanate to form solids.

A-3 REPORT

Report the appearance of the sample as clear liquid or hazy. If foreign particle present, report as foreign particle present.

ANNEX B

[*Table* 1, *Sl No.*(iii)]

DETERMINATION OF TOLUENE DIISOCYANATE CONTENT (ASSAY) BY GAS CHROMATOGRAPHY

B-1 PRINCIPLE

A sample of TDI is injected on a gas chromatographic capillary column and the results are calculated using the external standard method with area normalization.

B-2 APPARATUS

B-2.1 Gas Chromatograph, equipped with an FID detector, a split-splitless injector and a suitable electronic integrator/software.

B-2.2 Capillary Column, fused silica, with (5 percent-phenyl)-methyl polysiloxane phase with length 50 m; internal diameter 0.32 mm and film thickness 0.32 µm.

B-2.3 Syringe, 10 µl.

B-2.4 Analytical Balance, with least count of 0.1 mg.

B-3 REAGENT

B-3.1 80 : 20 Toluene Diisocyanate.

B-3.2 *m*-phenylene Diisocyanate.

B-3.3 1,2,4-Trichlorobenzene and 1,2,3-Trichlorobenzene.

B-3.4 *o*-Dichlorobenzene.

B-4 SAMPLING

Take a clean and dry 500 ml glass bottle. Do not expose the sample to moisture. Moisture reacts with toluene diisocyanate to form solids.

B-5 GAS CHROMATOGRAPH SETTINGS

Carrier gas	Hydrogen / Nitrogen / Helium
Pressure at head of column	151.685 kPa (22 psi)
Fuel gas and flowrate	Hydrogen : 40 ml, Air : 400 ml
Make up flow	As per the requirement of instrument make/model.
Sample preparation	Direct (as such sample)
Injection volume	1 µl
Split flow	125 ml /min
Detector	FID

Temperature programme of oven, detector and injector are given below:

Injector Temperature (°C)	*Detector Temperature (°C)*	*Oven*		
		Temperature (°C)	*Hold Time (min)*	*Ramp Rate (°C/min)*
250	250	125	2	6
		210	1	20
		240	10	0

NOTE — The above gas chromatographic (GC) conditions are suggestive. However, any GC method having difference in detector, column packing material and type (like packed / capillary, diameter, length, film thickness etc.), calibration technique (internal standard, external standard, area normalization, percent area etc.), carrier gas (He, H_2, N_2) may be used with applicable GC operating parameters, provided standardization and calibration of the components is established after setting GC parameters for the resolution and accuracy level as specified in this standard.

B-6 CALIBRATION

Prepare a standard solution containing the components given below, in the expected concentration range. A typical example of standard solution is given below for reference.

Component	*Mass, g*	*Concentration in percent (w/w)*
TDI	70.000 0	99.857 3
o-dichlorobenzene	0.025 0	0.035 7
m-phenylene diisocyanate	0.025 0	0.035 7
1,2,4-trichlorobenzene	0.025 0	0.035 7
1,2,3-trichlorobenzene	0.025 0	0.035 7

Make sure the GC instrument is adjusted to the conditions stated as above. Inject 1.0 µl of the standard at least three times and obtain the chromatograms. Calculate the calibration correction factor for each component by dividing its concentration by the respective area in its chromatogram. Find the arithmetic mean of the factors calculated for each component. Each factor is calculated to 4 decimal places. When using the factors, the calculation of the concentration of the sample is done using the mean obtained or use suitable software for auto calibration with respect to mass of calibration standard.

B-7 CALCULATION

B-7.1 Sum the areas of all unknown components.

B-7.2 Multiply the area of each component of the sample by its relative response correction factor as mentioned in **B-6**. Obtain the sum of all corrected areas. Normalize each corrected area to 100 percent and report each as mass percent or use suitable software for auto calculation with respect to calibration standard.

NOTE — Use response correction factor = 1, for the unknown components.

ANNEX C

[*Table* 1, *Sl No.* (iv)]

DETERMINATION OF HYDROLYZABLE CHLORINE

C-1 GENERAL

This method is used for the determination of hydrolyzable chlorine content of refined 2,4-toluene diisocyanate, 2,6-toluene diisocyanate and mixture of these toluene diisocyanates. The method is applicable to the determination of hydrolyzable chlorine content in the range of 0 to 0.10 percent.

C-2 PRINCIPLE

The isocyanate containing hydrogen chloride, carbonyl dichloride (CDC), carbamyl chlorides, aromatic acid chlorides and chloromethyl compounds is reacted with an aqueous methanol solution to convert the organic chloro compounds to free hydrochloric acid. The liberated HCl is then titrated with silver nitrate solution, potentiometrically.

C-3 SAMPLING

Take a clean and dry 500 ml glass bottle. Do not expose the sample to moisture. Moisture reacts with toluene diisocyanate to form solids.

C-4 APPARATUS

C-4.1 Magnetic Stirrer

C-4.2 Titrator, with a silver electrode versus an Ag/AgCl reference electrode or combine silver ring electrode.

C-4.3 Analytical Balance

C-4.4 Hotplate

C-5 REAGENTS

C-5.1 Methanol

C-5.2 Silver Nitrate, 0.0141 N standard

Dissolve 2.395 g silver nitrate in distilled water and make up to 1.0 l.

C-5.3 1 : 1 Nitric Acid

Take 1 volume of 70 percent nitric acid (HNO_3) and 1 volume of distilled water.

C-6 PROCEDURE

Weigh 13.5 ± 1.5 g of sample using any glass sampling device into a clean, dry 400 ml beaker. Add 125 ml methanol using graduated measuring cylinder and agitate on the magnetic stirrer. When the reaction starts, which is usually within a few seconds, slowly add 100 to 125 ml of chloride free distilled water. Cover with a clean watch glass and agitate for 10 to 15 min. Transfer the beaker from the stirrer to a hot plate digest the content at approximately 65°C to 70°C for about 30 min. Wash down the sides of the beaker with distilled water and cool to room temperature by means of an ice bath. Add 10 ml of 1 : 1 nitric acid to the sample. Titrate the sample with 0.0141 N silver nitrate using automatic titrator. From the instrument display, determine the volume of the solution used. Run blanks exactly in a same manner without adding sample.

C-7 CALCULATION

$$\text{Hydrolyzable chlorine, percent} = \frac{(A-B) \times N \times 3.546}{S}$$

where

A = volume silver nitrate, or sample titration, ml;

B = volume silver nitrate for blank titration, ml;

N = normality of silver nitrate solution; and

S = sample weight, g.

ANNEX D

[*Table* 1, *Sl No.* (v)]

DETERMINATION OF ACIDITY

D-1 PRINCIPLE

The isocyanates are reacted with excess *n*-propanol to form urethanes. The acidic components released are absorbed in the reaction mixture and titrate potentiometrically with methanolic potassium hydroxide.

D-2 SAMPLING

Take a clean and dry 500 ml glass bottle. Do not expose the sample to moisture. Moisture reacts with toluene diisocyanate to form solids.

D-3 APPARATUS

D-3.1 Automatic Titrator, complete with reference and glass electrode for *p*H measurement.

D-3.2 Buffer Solutions, *p*H 4 and 7.

D-3.3 Beaker, 250 ml.

D-4 REAGENTS

D-4.1 Potassium Hydroxide, 0.01 N methanolic, standardized to ± 0.000 1 N

Take 0.66 g potassium hydroxide (85 percent) in dry 1.0 l volumetric flask and dilute it to the mark with methanol. Stir thoroughly for 15 min.

D-4.2 Hydrochloric Acid, 0.01 N, standardized

Take 1.042 g hydrochloric acid (35 percent) in dry 1.0 l volumetric flask and dilute it to the mark with methanol. Stir thoroughly for 15 min.

D-4.3 *n*-Propanol.

D-5 PROCEDURE

Prepare the automatic titrator according to its operation manual. If necessary, standardize instrument to *p*H 4 with buffer solution. Take 100 ml of *n*-propanol in a 250 ml glass beaker and dip the combined glass electrode~~s~~ in n-propanol. Adjust its *p*H to 5.0 with either 0.01 N alcoholic potassium hydroxide or 0.01 N hydrochloric acid carefully. Remove electrodes from the solution. Add exactly 10 ml of the toluene diisocyanate sample into beaker using volumetric pipette while agitating the content. Cover with a watch glass and continue agitation for 10 min. Immerse electrodes in sample solution and titrate with 0.01 N alcoholic potassium hydroxide, till the endpoint is reached. The equivalence point will occur in the range of about 5.50 to 7.00 *p*H units.

D-6 CALCULATION

Acidity (*as* HCl), percent by mass

$$= \frac{A \text{ x } N \text{ x } 3.647}{10 \text{ x density of material } (1.22)}$$

where

A = volume of potassium hydroxide solution required, ml; and

N = normality of potassium hydroxide solution

D-7 Report results to nearest 0.000 1 percent.

ANNEX E

[*Table* 1, *Sl No.* (vi)]

DETERMINATION OF TOTAL CHLORINE

E-1 PRINCIPLE

The organic matter in the sample is destroyed by combustion with oxygen, converting the organically combined chlorine to ionic chloride. The concentration of chloride ion is then determined by potentiometric titration with dilute standard silver nitrate solution.

E-2 APPARATUS

E-2.1 Oxygen Combustion Bomb (Fig 1) **and Ignition Unit** (Fig 2)

E-2.2 Automatic Titrator

E-2.3 Electrode, reference and silver indicating or combine silver ring electrode.

E-2.4 Micro Burette, 5 ml capacity, 0.01 ml graduation.

E-2.5 Weighing Bottle, suitable for weighing a liquid sample by difference to the nearest 0.5 mg.

E-2.6 Nichrome Fuse Wire, for ignition.

E-3 REAGENTS

E-3.1 Sodium Carbonate, 10 percent aqueous solution

Take 100 g sodium carbonate powder in a 1.0 l volumetric flask and make it up to the mark with distilled water.

E-3.2 Oxygen, pure grade

E-3.3 1 : 1 Nitric Acid (HNO_3)

Take 1 volume of 70 percent HNO_3 and 1 volume of distilled water

E-3.4 Silver Nitrate ($AgNO_3$), 0.014 1 N standard

Dissolve 2.395 g silver nitrate in distilled water and make it up to 1.0 l.

E-3.5 Ethyl Alcohol, absolute (free from chloride)

E-3.6 Methyl Red Indicator

Take 0.1 g methyl red powder and dissolve it in 100 ml ethanol.

E-4 SAMPLING

Take a clean and dry 500 ml glass bottle. Do not expose the sample to moisture. Moisture reacts with toluene diisocyanate to form solids.

E-5 PROCEDURE

Check to be sure that the combustion bomb, oxygen lines, and fittings are free of oil and grease. Small quantities of either may cause violent rupture of the equipment. Check to be sure that the combustion bomb, oxygen lines and fittings are not leaking. Weigh 0.9 to 1.0 g (accurately up to 0.000 5 g) sample using any glass sampling device into the combustion crucible. Add 1 to 2 ml of ethyl alcohol in it. Fit a 10 cm length of nichrome wire on to the two electrodes as mentioned in figure. Put the crucible in place on the loop electrode and adjust the fuse wire under dip of the sample in the crucible but wire does not touch the crucible at any surface. Take 10 ml 10 percent sodium carbonate solution in the combustion bomb. Carefully put the bomb head in the bomb cylinder, the contact ring on top of the bomb head and screw tight. Close the outlet valve of combustion bomb. Attach the union on the oxygen filling connection to the inlet valve of the combustion bomb. Pass oxygen slowly until 20-25 atmosphere pressure of oxygen gets built in the combustion bomb. Connect the ignition terminal on the top of the combustion bomb. Insert the plug to the ignition unit and ignite combustion in combustion bomb by pressing the button of ignition unit. After ignition, allow the bomb to cool for 10 to 15 min. Release the pressure very slowly, for about 5 min. When all the oxygen pressure has been released, rinse the head and outside of the combustion bomb with distilled water. Open the head of combustion bomb from cylinder, carefully wash the bomb head, the electrodes, crucible and the cylinder walls with distilled water. The total volume of washings is normally about 75 ml. Transfer the washing into a 250 ml conical flask / glass beaker. Add 2-3 drops of methyl red indicator and add 1 : 1 nitric acid (HNO_3) till color changes from yellow to red. Add 75 ml of ethanol to cover the electrode tips and cool the solution to 30°C. Titrate against 0.014 1 N silver nitrate ($AgNO_3$) till endpoint which is determined potentiometrically. Note down the volume of the solution used and calculate the result by using the following formula. Run a blank on all reagents (including oxidation) and obtain a titer value for the blank.

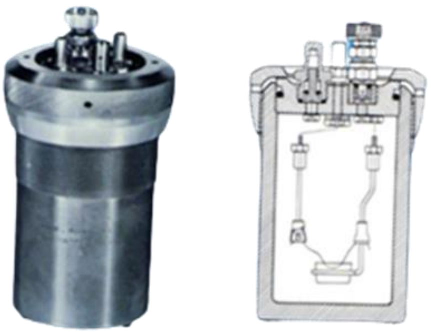

FIG. 1 OXYGEN COMBUSTION BOMB

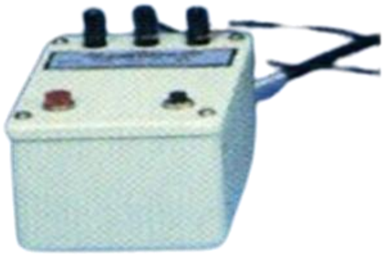

FIG 2 IGNITION UNIT

E-6 CALCULATION

Total Chlorine, percent by mass

$$= \frac{(A-B) \text{ x } N \text{ x } 3.55}{S}$$

where,

A = volume of silver nitrate required to titrate sample, ml;

B = volume of silver nitrate required to titrate blank, ml;

N = normality of the silver nitrate solution; and

S = mass of sample, g.

E-7 Report results to nearest 0.005 percent.

ANNEX F

[*Table* 1, *Sl No.* (vii)]

DETERMINATION OF ISOMER RATIO

F-1 GENERAL

Two methods namely, Method A Determination of isomer ratio by infrared spectroscopy and Method B Determination of isomer ratio by gas chromatography are prescribed. In case of disputes, method A determination of isomer ratio by infrared spectroscopy shall be the referee method.

F-2 METHOD A

F-2.1 General

This method is applicable to samples of TDI containing 17 to 25 percent of the 2,6-TDI isomer and 75 to 83 percent of the 2,4-TDI isomer.

F-2.2 Principle

This method is based on a quantitative intensity measurement of absorption bands arising from out-of-plane C-H deformation vibrations of the aromatic ring (12.35 mm and 12.8 mm). Since the system shows apparent deviations from beer´s law, it is necessary to construct calibration curves for the procedure. The method is carried out by dissolving the sample in cyclohexane and running the spectrum in the 12 to 13 mm region (840 nm to 760 nm), measuring the absorbance, calculating the absorbance ratio and converting the value obtained to a corresponding mass composition by the use of a previously established equation or calibration curve.

F-2.3 Sampling

The sample shall be taken in a clean, dry, 500 ml glass bottle. Do not expose the sample to moisture. Moisture reacts with toluene diisocyanate to form solids.

F-2.4 Apparatus

F-2.4.1 *IR spectrophotometer*

Any single or double beam infrared (IR) spectrophotometer having sufficient resolution to distinguish between the two peaks of the 2,4-TDI isomer doublet at 810 nm is adequate for use with this method.

F-2.4.2 *Cells*

Sodium chloride (NaCl) sealed liquid absorption cells having a thickness of 0.2 mm are required. The actual thickness of the cells should be known to within ± 0.002 mm.

F-2.4.3 *Volumetric flasks*, 25 ml, glass stoppered.

F-2.4.4 *Vials*, about 10 ml, also glass stoppered.

F-2.5 Reagents

F-2.5.1 *Cyclohexane***,** this solvent should be stored over some adequate desiccant to remove traces of moisture (recommended desiccants are : silica gel and molecular sieve type 5A).

F-2.5.2 *Diisocyanate standards*, certified reference material of pure 2,4-toluene diisocyanate and 2,6-toluene diisocyanate.

NOTE — Since these diisocyanate will react with moisture and may discolor in the presence of air, they should be stored and handled under dry nitrogen.

F-2.6 Calibration

F-2.6.1 Take six dry, 10 ml glass stoppered flasks and prepared a calibration blend by weighing into them the amounts of pure 2,4-TDI and 2,6-TDI as given in the following table:

2,4-TDI, percent	*2,6-TDI, percent*	*2,4-TDI, g*	*2,6-TDI, g*
75	25	2.6250	0.8750
77	23	2.6960	0.8050
79	21	2.7650	0.7350
80	20	2.8000	0.7000
81	19	2.8350	0.6650
83	17	2.9050	0.5950

F-2.6.2 Swirl each flask to mix its contents very well. Now weigh 1.000 0 g of each of the above standards into dry 25 ml volumetric flasks. Dilute to volume with dry cyclohexane and mix thoroughly. Using the 0.2 mm sealed liquid absorption cells, fill both the reference and sample cells with cyclohexane and record the solvent spectrum from 840 nm to 760 nm three times. Empty the sample cell and fill it with the standard solution. Record the spectrum superimposing it over the previously recorded solvent spectrum. Repeat steps for each standard solution. Measure the absorbance of each of the calibration solutions at the following wavelengths, starting from the solvent blank absorption spectrum: 2,4-TDI at 810 nm and 2,6-TDI at 782 nm

F-2.6.3 Calculate now the absorbance ratios and the mass ratios as follows:

Absorbance ration =

$$\frac{Absorbance\ of\ 2{,}4{-}TDI\ at\ 810\ nm}{Absorbance\ of\ 2{,}6{-}TDI\ at\ 782\ nm}$$

Mass ratio =

$$\frac{(Mass\ of\ 2{,}4-TDI\ taken\ for\ blend\ preparation)}{(Mass\ of\ 2{,}6-TDI\ taken\ for\ blend\ preparation)}$$

F-2.6.4 Obtain a calibration curve by plotting the absorbance ratios on the y-axis versus the mass ratios of 2,4-TDI to 2,6-TDI in each solution on the x-axis.

F-2.7 Procedure for Samples of 80/20 TDI

F-2.7.1 Weigh 1.000 0 g of the TDI sample using lung ray pipette or any glass sampling device into a dry 25 ml glass stoppered volumetric flask. Dilute to volume with dry cyclohexane and mix thoroughly. Fill two 0.2 mm cells with pure solvent and obtain its spectrum from 840 to 760 nm. Fill 0.2 mm cell of IR spectrophotometer with sample and take the spectrum of sample and note down the absorbance at 810 nm and 782 nm. Take a blank run of cyclohexane solvent and subtract the absorbance of respective wavelength from sample run absorbance.

F-2.8 Calculation

F-2.8.1 Absorbance ratio of 2, 4-TDI and 2,6-TDI=

$$\frac{(Sample\ absorbance\ at\ 810\ nm)-(Blank\ absorbance\ at\ 810\ nm)}{(Sample\ absorbance\ at\ 782\ nm)-(Blank\ absorbance\ at\ 782\ nm)}$$

F-2.8.2 Obtain the mass ratio using calibration curve of Absorbance ratio vs Mass ratio as per **F-2.6.4.**

F-2.8.3 Calculate 2,4-TDI percent and 2,6-TDI percent as bellow:

2,6-TDI percent = 100 / (R+1)

2,4-TDI percent = 100 – (2,6-TDI percent)

where

R = Mass ratio found out as per **F-2.8.2**.

F-3 METHOD B

F-3.1 APPARATUS

F-3.1.1 Gas Chromatograph, equipped with an FID detector, a split-splitless injector and a suitable electronic integrator/software.

F-3.1.2 Capillary Column, fused silica, with (5 percent-phenyl)-methyl polysiloxane phase with length 50 m; internal diameter 0.32 mm and film thickness 0.32 µm.

F-3.1.3 Spoid, 3 ml.

F-3.1.4 Snap cover glasses, glass vial of 20 ml (for auto sampler).

F-3.1.5 Analytical Balance, with least count of 0.1 mg.

F-3.1.6 Syringe, 10 µl.

F-4 REAGENTS

F-4.1 Toluene

F-4.2 Diisocyanate Standards, certified reference material of pure 2,4-toluene diisocyanate and 2,6-toluene diisocyanate.

F-5 GAS CHROMATOGRAPH SETTINGS

Carrier gas	Nitrogen
Pressure at head of column	117.211 kPa (17.0 psi)
Fuel gas and Flow rate	H_2 : 40 ml/min and Air : 400 ml/min
Make up flow	30 ml/min or depends on make and model of GC
Sample preparation	Take 0.4 g sample and mix with 10 ml toluene.
Injection volume	1 µl

Split flow	109 ml/min
Detector	FID

Temperature programme of oven, detector and injector are given below:

Injector Temperature (°C)	*Detector Temperature (°C)*	*Oven*		
		Temperature (°C)	*Hold Time (min)*	*Ramp Rate (°C/min)*
250	300	150	8	10
		230	3	—

NOTE — The above gas chromatographic (GC) conditions are suggestive. However, any GC method having difference in detector, column packing material and type (like packed / capillary, diameter, length, film thickness etc.), calibration technique (internal standard, external standard, area normalization, percent area etc.), carrier gas (He, H_2, N_2) may be used with applicable GC operating parameters, provided standardization and calibration of the components is established after setting GC parameters for the resolution and accuracy level as specified in this standard.

F-6 CALIBERATION

The response correction factors are derived from the TDI samples with an exactly known ratio, approximately 80 : 20 of 2,4-TDI to 2,6-TDI. Take approximately 0.8 g of 2,4-TDI and 0.2 g of 2,6-TDI and mix them. The mixture is then examined by the gas chromatography method according to the procedure (**F-7**). Calculate the calibration correction factor for each component by dividing its concentration by the respective area in its chromatogram.

The purity of the substances is to be considered at the calculation of the response factors. For example, the content of 2,4-TDI will be determined by a separate analysis of the 2,4-TDI as area percent. Then correct the weigh-input of the 2,4-TDI in the factor solution. Do the same with the 2,6-TDI.

F-7 PROCEDURE

Pipette 0.4 g of sample into a snap cover glass. Add 10 ml of toluene to it. The mixture is then homogenized. Take approximately 1.5 ml of the homogenized solution and pour it into GC vial. Place vial in autosampler and start analysis or inject manually 1.0 µl sample using microliter syringe.

F-8 CALCULATION

F-8.1 2,4-TDI and 2,6-TDI content is calculated as

2,4-TDI, percent by mass =

$$\frac{A_{2,4\text{-}TDI} \times F_{2,4\text{-}TDI}}{A_{2,4\text{-}TDI} \times F_{2,4\text{-}TDI} + A_{2,6\text{-}TDI} \times F_{2,6\text{-}TDI}}$$

where

$A_{2,4\text{-}TDI}$ = peak area of 2,4-TDI;

$F_{2,4\text{-}TDI}$ = correction response factor for 2,4-TDI;

$A_{2,6\text{-}TDI}$ = peak area of 2,4-TDI; and

$F_{2,6\text{-}TDI}$ = correction response factor for 2,4-TDI

2,6-TDI content is calculated by the difference of 2,4-TDI content from 100 percent.

or

Use suitable software for auto calculation with respect to calibration standard.

F-8.2 Report isomer ratio as the ratio of 2,4-TDI content to 2,6-TDI (2,4-TDI : 2,6-TDI)

ANNEX G

(*Informative Annex*)

G-1 SAFETY INSTRUCTIONS TO BE TAKEN DURING TESTING

G-1.1 General Safety Instructions

a) Avoid skin contact and do not inhale fumes.

b) Use adequate ventilation to keep the vapor concentration at a minimum.

c) Any spills of nitric acid and sulphuric acid must be washed off immediately with cold water.

d) Use rubber gloves, an apron, and goggles when handling these reagents.

e) Methanol and ethanol are flammable. Keep away from sparks and flames

f) Avoid skin contact of mercury. If any spillage, use the vacuum trap to suck up spills immediately and transfer to the bottle kept for collection of discarded mercury.

G-1.2 Safety Instructions for use of Oxygen Combustion Bomb

Operation of oxygen combustion bomb must be done following utmost safety precautions.

a) The bomb must be pressure tested periodically at 13.789 51 MPa (200 0 psi).

b) The pressure gauge must be tested periodically for accuracy at the pressure used in the bomb [3.10264 MPa (450 psi)].

c) It also should withstand the pressure permitted by the safety disc [4.82633 MPa (700 psi)].

d) Before admitting oxygen to the bomb, make sure that the bomb including the valve packing and the oxygen line with fittings are free of oil and grease to prevent a violent rupture of the equipment.

ANNEX H

(*Foreword*)

COMMITTEE COMPOSITION

Organic Chemicals, Alcohols and Allied Products Sectional Committee, PCD 9

Organization(s)	*Representative(s)*
Chemical Engineering and Process Development Division, NCL, Pune	DR C. V. RODE ***(Chairman)***
Alkyl Amines Chemicals Limited, Mumbai	SHRI S. V. NIKUMBHE SHRI SAMEER KATDARE *(Alternate)*
All India Alcohol-Based Industries Development Association (AABIDA), Mumbai	SHRI K. L. RAPHAEL SHRI KIRTI GAJJAR (*Alternate)*
All India Distillers Association (AIDA), New Delhi	SHRI V. N. RAINA
BASF India Limited, Mumbai	SHRI KIRAN BHAT SHRI HEMAL (*Alternate)*
CSIR- Central Drug Research Institute (CDRI), Lucknow	DR SANJEEV KANOJIYA
Chemical And Petrochemicals Manufacturers Association (CPMA), New Delhi	SHRI MAHINDER SINGH
Deepak Fertilizer, New Delhi	DR L. B. YADAWA SHRI SURESH AMLE (*Alternate)*
Deepak Phenolics Limited, Vadodara	SHRI DHARMESH SIDDHAPURIA SHRI SANDIP KUMAR PANDYA (*Alternate)*
Dow Chemical International Private Limited, Mumbai	SHRI V.MOHANDOSS SHRI GOVIND GUPTA (*Alternate)*
Godavari Biorefineries, Mumbai	SHRI SHANUL LAXMANRAO PAGAR SHRI APPASAHEB J. WANI (*Alternate)*
Gujarat Narmada Valley Fertilizers Company Limited, Gujarat	SHRI R. M. PATEL SHRI C. S. PATEL (*Alternate*)
Hindustan Organic Chemicals Limited (HOCL), Mumbai	SHRI DELEEP KUMAR. K SHRI V. MOHAN (*Alternate)*
India Glycols Limited, Uttarakhand	SHRI S. R. SONI SHRI ALOK SINGHAL (*Alternate*)

Organization(s)	*Representative(s)*
Indian Chemical Council (ICC), Mumbai	Dr Mritunjay Chaubey Shri J Sevak (*Alternate)*
Jubilant Life Sciences Limited, Uttar Pradesh	Shri Hari Mohan Lohani
Laxmi Organic Industries, Mumbai	Shri J. P. Suryavanshi Dr Vijay S. Mishra (*Alternate)*
Department of Chemicals & Petrochemicals, Ministry of Chemicals & Fertilizers, New Delhi	Shri O. P. Sharma Shri Varun Singh Poonia (*Alternate)*
National Chemical Laboratory (NCL), Pune	Dr Ravindar Kontham Dr Udaya Kiran Marelli (*Alternate)*
National Test House (NTH) , Ghaziabad	Shri Debashis Saha Dr Gopal Krishan (*Alternate)*
Reliance India Limited (RIL), Mumbai	Shri K. K. Sreeramachandran Shri Vasant Warke (*Alternate)*
United Phosphorus Limited (UPL), Mumbai	Shri M. D. Vachhani
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BIS Directorate General,	Shrimati Nagamani. T, Scientist 'E' And Head (Pcd) [Representing Director General (*Ex-Officio*)]

Member Secretary

Ms Aditi Choudhary

Scientist 'B', BIS

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